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A RAPID CHEMICAL ANALYSIS OF THE INGREDIENTS OF NOL-130 PRIMER MIX

STANLEY SEMEL

DECEMBER 1979

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
LARGE CALIBER
WEAPON SYSTEMS LABORATORY
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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

A method has been developed for the rapid chemical analysis for the ingredients of NOL-130 primer mix, utilizing Differential pulse polarography. The sample is dissolved in a hydrochloric acid solution and is injected directly into an electrolyte solution and polarographed without any additional separation or handling. Lead styphnate, lead azide, tetracene, and antimony sulfide can be analyzed directly, barium nitrate is obtained by difference. The NOL-130 can be analyzed in 30 minutes.

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INTRODUCTION

The Office of the Project Manager, Munitions Production Base Modernization and Expansion, Dover, NJ, requested that a rapid method of chemical analysis be developed for the ingredients of NOL-130 primer mix, prior to loading detonators. Although the method of analysis listed in Military Specification, Detonator, Stab, M55, MIL-D-14978A is simple, it is time-consuming and will not meet the thirty minute requirement for a modern Government Owned Company Operated (GOCO) Ammunition Plant.

There are other methods of chemical analysis for the ingredients of NOL-130 primer mix; however, only an instrumental method of analysis will meet the time constraint. The following instrumental methods were considered: X-ray fluorescence for lead, antimony and barium; 14 Mev neutron activation for nitrogen and oxygen; atomic absorption for lead, antimony, and barium; combustion methods for carbon, nitrogen, oxygen, and hydrogen; and polarography for lead, antimony, tetracene, and styphnate. From the instrumental methods of analysis listed above, polarography was selected as a single method of analysis since it works equally well for metals, non-metals, and organics.

Earlier work by Lingane, et al. (ref. 1) indicated the feasibility of polarographic analysis for some of the ingredients of primers. A typical mixture analyzed by Lingane was composed of potassium chlorate, copper (I) thiocyanate, antimony sulfide, lead azide, and mercury fulminate. Wild (refs. 2,3) developed a method for tetracene based on its dissolution in hydrochloric acid to give a well-defined reduction wave at the dropping mercury electrode. He also developed a method for the polarographic estimation of tetracene and nitro-resorcinates in a single cap. Leake and Reynolds (ref. 4) determined both lead and styphnic acid in the same solution with recoveries better than ± 5%.

Ribaudo and Cragle (ref. 5) developed a polarographic analysis of a primer mix for the M60 base-detonating fuze. The mix consists of 2,4,6-trinitrotoluene, antimony sulfide, lead thiocyanate and potassium chlorate; all ingredients were determined polarographically except potassium chlorate, which was determined by difference. The standard deviations of all the ingredients are 0.1% or better. The actual working time for the three determinations is 1.5 hours, and the overall time of analysis is 2.5 hours. Currently polarography is being used by Witnack (ref. 6) for the determination and monitoring of ordnance-derived pollutants. Based on the previous work cited, the feasibility of polarographic techniques for explosives has been demonstrated and a method developed for the analysis of ingredients of NOL-130 primer mix.

EXPERIMENTAL PROCEDURE

Composition of NOL-130

The nominal composition of NOL-130 primer mix, as described in MIL-D-14978, consists of:

	Percent
Lead azide	20.0 ± 2.0
Basic or normal lead styphnate	40.0 ± 2.0
Tetracene	5.0 ± 0.5
Barium nitrate	20.0 ± 2.0
Antimony sulfide	15.0 ± 1.5

Sample Preparation

After carefully mixing the NOL-130, weigh accurately 0.100 g and place it in a 300 mL beaker containing a magnetic stirring bar. Place the beaker on a magnetic stirrer in a fume hood behind a barricade and carefully add 50 mL of concentrated hydrochloric acid[1]. Stir the acidified sample for five minutes, add 50 mL of distilled water and continue stirring for two minutes. At this point, add an additional 50 mL of concentrated HCl and continue stirring for another three minutes. Add an additional 50 mL of distilled water, bringing the total volume to 200 mL and stir for an additional two minutes. After the 12 minutes of stirring, check the solution for clarity. If a white solid is still present, add an additional 10-15 mL of distilled water and stir until the solution is clear. Quantitatively transfer the solution, with the aid of a wash bottle, to a 250 mL volumetric flask. Dilute to volume with additional distilled water. The flask is then stoppered and mixed by inverting several times until the solution is clear. Cool the flask to room temperature and check the total volume. The solution is now ready for analysis.

A synthetic sample of NOL-130 was prepared by carefully weighing the dry ingredients in the proper proportion.

Instrumentation

Polarographic data were obtained with a model 374 Polarographic Analyzer (Princeton Applied Research Corporation, Princeton, NJ) and a model 303 Static Mercury Drop Electrode. For this work, the dropping

[1] At this point, HN_3 and H_2S are evolved.

mercury electrode was used. As a reference electrode, a saturated solution of KCl/AgCl was employed. The counter electrode furnished with this model, is a 5.2 cm long, 0.1 cm diameter platinum wire.

The samples were analyzed by Differential Pulse Polarography using the programming wave form (fig. 1). In this technique, a staircase ramp is applied with a fixed height pulse super-imposed on the ramp near the end of the life of each mercury drop. The current is sampled twice, just before the application of the pulse and again at the end of the pulse (5-100 mV). The difference between the current values is inputed as the current signal. To a close approximation, the difference current varies as the derivative of the polarographic wave, giving a peak output presentation as a peak shape. The polarogram is a plot of current difference versus applied potential.

Instrumentation Settings for the Model 374 Polarograph

Analytical Technique		Differential Pulse Polarography
Scan rate	-	Slow
Replications	-	One
Sensitivity	-	Medium

4M LiCl electrolyte for antimony, tetrace and lead	0.1 M Tetra-n-butylammonium hydroxide electrolyte for styphnate
Initial potential - 0.050V	Initial potential - 0.600V
Final potential - 0.510V	Final potential - 0.900V

Preparation of Supporting Electrolytes

1. 4M Lithium Chloride (LiCl) Certified Reagent or equivalent:

Dissolve 169.56 g of LiCl in 1000 mL of distilled water. Stir until solution is complete. Cool to room temperature before using.

2. 0.1 M Tetra-n-butylammonium hydroxide:

Dissolve 100 mL of 1.0 M tetra-n-butylammonium hydroxide[2] (polarographic grade) in 900 mL distilled water. Stir the solution and cool to room temperature. Blank curves must be run on both electrolytes to see that there are no interferring peaks in the area of any ingredient of the NOL-130 primer mix

[2] Southwestern Analytical Chemicals, Inc., Austin, TX, or equivalent.

Blank Curve Determination

With the aid of a volumetric pipette, transfer 10 mL of the electrolyte solution to a borosilicate glass polarographic cup. Place the cup in the analysis position of the model 303 Mercury Drop Electrode, set the polarograph for blank determination and pass oxygen-free nitrogen or argon gas through the solution for 5 minutes. Record the polarogram. At this point, there should be little or no background interference due to the electrolyte solutions. This blank will now be stored in the polarograph and will be subtracted from the standard and sample curves. Two electrolytes, 4M LiCl and 0.1 M tetra-n-butylammonium hydroxide are polarographed as indicated under "Analysis of Unknown Samples."

Standard Curve Determination

Obtain a sample of NOL-130 that has been analyzed previously to use as a standard for this analysis. Prepare the standard as indicated under "Sample Preparation". Set the polarograph for standard, with the aid of an Eppendorf pipette, add 100 µl of the standard solution to the blank, pass oxygen-free nitrogen or argon through the sample for 0.5 minutes and run the standard. At the end of the run, press "Playback" and then "Calculate". The polarograph will print out the initial potential, the half-wave potential at each peak, the concentration at each peak in nano-amps of current, the date, sample number, if desired, and final potential. The standard concentration will then be stored in the memory. This technique provides the microprocessor with the data it needs to do the concentration calculations of the unknown samples and must be done before the analysis of the unknown samples.

Analysis of Unknown Samples

After the blank and standard samples are run, the unknown sample is ready to be run. The sample-indicating light on the instrument panel should be lit. Place 10 mL of the appropriate electrolyte in another polarograph cup and set the instrument as indicated under "Instrument Settings" for the analysis of NOL-130. For the determination of antimony, tetracene, and lead, use 4M LiCl and for styphnate, 0.1M tetra-n-butylammonium hydroxide. With the aid of an Eppendorf pipette (or equivalent) add exactly 100 µl of the unknown sample solution as prepared under "Sample Preparation" and pass oxygen-free nitrogen or argon gas through the sample for 5 minutes. Run the sample exactly as the standard determination. At the end of each run, the microprocessor will print out the concentrations in ppm contained in the unknown for each ingredient.

Calculate the percent of each ingredient as follows:

$$\%\ \text{(Normal) lead styphnate} = \frac{\text{Conc. normal styph (ppm)} \times 1.7944}{4}$$

$$\%\ \text{(Basic) lead styphnate} = \frac{\text{Conc basic styph (ppm)} \times 2.4962}{4}$$

$$\%\ \text{Lead azide (normal lead styphnate)} = \frac{[\text{Conc. Pb (ppm)} - (\text{Conc. styph (ppm)} \times .7930)] \times 1.405}{4}$$

$$\text{(basic lead styphnate)} = \frac{[\text{Conc. Pb (ppm)} - (\text{Conc. styph (ppm)} \times 1.4981)] \times 1.405}{4}$$

$$\%\ \text{Tetracene} = \frac{\text{Conc. tetracene (ppm)}}{4}$$

$$\%\ \text{Antimony sulfide} = \frac{\text{Conc. Sb (ppm)} \times 1.395}{4}$$

% Barium nitrate = Add the four ingredients and subtract from 100, on a moisture-free basis

RESULTS AND DISCUSSION

The interference of the various ingredients in a mixture such as NOL-130 creates the difficulty in the analysis. Since the objective of this investigation was a rapid determination of the constituents, a method had to be developed for each ingredient, without the use of time-consuming separation techniques, which are normally recommended in polarography. The two electrolyte system was then developed, where three ingredients can be quantitatively determined and the fourth run in a second electrolyte solution. With the use of a second polarograph the four ingredients can be determined simultaneously. Figure 2 represents the differential pulse polarogram for the determination of antimony, tetracene, and lead. These three ingredients can be determined simultaneously in one polarogram. The half-wave potential for antimony is approximately -0.100 V. Tetracene has a half-wave potential at -0.250 V and lead at -0.416 V. However, these half-wave potentials will change with time and a standard must be run each day.

Styphnate yields three reduction half-wave potentials which vary considerably with the pH and specific electrolyte. In figure 3, the peak at -0.744 in 0.1 M tetra-n-butylammonium hydroxide was used since no other NOL-130 ingredient interferred.

The preparation and analysis of a synthetic NOL-130 sample was in accordance with the procedure described in the Experimental section. At the request of the Product Assurance Directorate, ARRADCOM, fifteen determinations were made and the results are reported in table 1. Samples of three different blends, 6, 13, and 15 of Batch 19 of plant-manufactured NOL-130 primer mix, were received from Lone Star Army Ammunition Plant and analyzed as above. A total of ten determinations of each blend was done and one reported in table 2. A total of three batches, nos. 53, 54, and 56 was anyalzed in accordance with MIL-D-14978A dated 30 October 1972, at the Lone Star AAP. These samples were also analyzed by the polarographic method. The results of these analyses, together with those of Lone Star AAP, are reported in tables 3, 4, and 5. These results were obtained independently at ARRADCOM and Lone Star laboratories.

CONCLUSIONS

The results listed in tables 1 and 2 indicate that the polarographic method of analysis meets the specification requirements as indicated by the standard deviations of the individual ingredients. The comparison of the specification method and the polarographic method indicate that these methods of analysis are equivalent as shown by the standard deviations listed in tables 3, 4, 5. The time of analysis meets the 30 minute requirement.

RECOMMENDATIONS

It is recommended that the differential pulse polarographic method of analysis for the ingredients of NOL-130 primer mix be adapted as a specification method of analysis since it is a more rapid and as equally accurate and precise a method as the existing specification method. It is also recommended that an atomic absorption spectrophotometric method be developed for the elements, lead, barium, and antimony as a complimentary method. The atomic absorption technique, in conjunction with the polarographic method will permit the simultaneous analysis of all the ingredients in NOL-130.

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6. G.C. Witnack, "Applied Polarography for Analysis of Ordnance Materials," Part 3, CA NWC-5860, Naval Weapons Center, China Lake, CA, January 1979.

Table 1. Polarographic determination of NOL-130 ingredients, synthetic mixture

Table 1. Polarographic determination of NOL-130 ingredients.

	Lead styphnate found %	Lead azide found %	Tetracene found %	Barium nitrate* found %	Antimony sulfide found %
1.	40.45	20.15	5.10	20.60	15.18
2.	40.49	20.20	5.20	20.50	14.96
3.	40.60	20.31	5.05	20.55	14.85
4.	40.39	20.50	5.22	20.44	15.31
5.	40.20	20.12	5.31	20.70	15.40
6.	40.55	19.92	5.01	20.66	14.79
7.	40.10	20.54	5.11	20.36	15.10
8.	39.90	20.51	5.35	19.79	15.05
9.	40.16	20.30	5.20	20.20	15.16
10.	40.61	20.16	5.16	20.59	15.13
11.	39.89	19.88	4.96	20.33	14.77
12.	39.98	20.13	5.16	20.41	15.04
13.	40.30	20.05	5.09	20.11	15.21
14.	40.11	20.09	5.16	20.37	15.10
15.	39.81	20.21	4.99	19.84	15.15
Mean	40.24	20.18	5.20	20.36	15.08
Standard deviation	±0.27	±0.20	±0.13	±0.15	±0.18
Added, %	40.00	20.00	5.00	20.00	15.00

* Barium nitrate was obtained by difference.

Table 2. Polarographic determination of Lone Star NOL-130, Batch 19

Table 2. Polarographic determination of Lone Star NOL-130, Batch 19

Det'n	Lead styphnate %	Lead azide %	Tetracene %	Antimony sulfide %	Barium nitrate* %
Blend 6					
1.	41.30	20.60	5.00	14.90	18.20
2.	41.41	20.41	5.22	15.00	18.23
3.	41.53	20.39	5.17	15.02	17.89
4.	41.22	20.81	5.25	15.11	17.61
5.	41.06	20.30	4.98	15.03	18.63
6.	40.98	20.41	5.16	15.19	18.26
7.	40.87	20.29	4.91	14.80	19.13
8.	41.22	20.85	5.11	15.20	17.62
9.	41.44	20.77	5.30	15.16	17.33
10.	40.85	20.91	5.01	15.15	18.08
Mean	41.19	20.57	5.04	15.06	18.10
Standard deviation	±0.24	±0.24	±0.13	±0.13	±0.53
Blend 13					
1.	40.10	20.10	5.08	14.88	19.84
2.	40.09	20.20	5.20	14.79	19.72
3.	40.62	19.78	5.01	15.00	19.59
4.	40.98	19.90	4.89	14.87	19.36
5.	40.33	20.05	4.93	14.92	19.77
6.	40.88	19.89	5.10	14.90	19.23
7.	40.40	20.30	5.09	14.81	19.40
8.	40.16	20.18	5.20	15.08	19.38
9.	40.02	20.17	5.00	15.20	19.61
10.	40.90	20.00	4.89	15.06	19.15
Mean	40.45	20.07	5.04	14.95	19.51
Standard deviation	±0.37	±0.16	±0.11	±0.13	±0.24
Blend 15					
1.	41.20	19.96	5.10	14.62	19.12
2.	41.08	19.19	5.20	14.53	20.00
3.	41.30	19.62	5.01	14.85	19.22
4.	41.00	19.96	5.25	14.39	19.40
5.	40.86	20.05	5.06	14.90	19.13
6.	40.70	19.95	5.29	14.75	19.31
7.	41.30	19.09	5.30	14.69	19.62
8.	41.10	19.16	5.10	14.79	19.85
9.	40.69	20.10	5.26	15.01	18.94
10.	41.00	19.88	5.04	14.48	19.60
Mean	41.02	19.70	5.16	14.70	19.32
Standard deviation	±0.22	±0.40	±0.11	±0.20	±0.34

*Barium nitrate was obtained by difference from 100%.

Table 3. NOL-130 Lone Star Batch 53.

	Lead styphnate		Lead azide		Tetracene		Antimony sulfide		Barium nitrate	
	Spec method	Polaro-graph	Spec method[a]	Polaro-graph	Spec method	Polaro-graph	Spec method	Polaro-graph	Spec method	Polaro-graph[b]
	39.49	40.21	19.89	19.96	5.48	5.06	15.00	14.86	20.14	19.91
	40.17	39.98	18.86	20.10	5.31	5.11	14.57	15.21	21.09	19.60
	40.62	39.56	18.14	20.42	5.50	4.99	14.83	15.06	20.91	19.97
	40.26	40.07	18.39	19.53	5.30	5.16	14.42	14.67	21.63	20.57
	39.48	41.04	18.77	19.16	5.17	5.23	14.41	14.80	22.17	19.77
	39.59	40.40	18.88	18.87	5.00	5.31	15.08	15.15	21.45	20.27
	41.36	40.62	19.12	18.63	5.39	4.89	14.77	14.73	19.36	21.13
	41.21	40.32	17.68	18.98	5.33	5.40	15.13	14.90	20.65	20.40
	40.61	39.97	18.57	19.02	5.17	5.22	14.58	14.59	21.07	21/22
	40.26	40.15	19.17	19.04	5.12	5.04	14.95	14.77	20.50	21.00
Mean	40.31	40.23	18.75	19.37	5.28	5.14	14.77	14.87	20.90	20.38
Standard deviation	±0.67	±0.40	±0.61	±.60	±0.16	±0.15	± 0.27	±0.21	± 0.80	±.58

[a]Lead azide was obtained by difference from 100%.

[b]Barium nitrate was obtained by difference from 100%.

Table 3. NOL-130 Lone Star Batch 53

Table 4. NOL-130 Lone Star Batch 54.

	Lead styphnate		Lead azide		Tetracene		Antimony sulfide		Barium nitrate	
	Spec method	Polaro-graph	Spec method[a]	Polaro-graph	Spec method	Polaro-graph	Spec method	Polaro-graph	Spec method	Polaro-graph[b]
	39.87	39.64	18.70	20.12	5.35	5.04	14.92	15.06	21.16	20.14
	40.68	40.12	18.82	19.95	5.01	5.10	14.76	14.83	20.73	20.00
	40.72	40.76	19.01	18.64	5.08	4.90	14.63	14.47	20.56	21.23
	41.53	40.79	19.12	19.20	5.22	4.85	14.41	14.61	19.72	20.55
	40.38	39.55	18.54	19.10	5.20	5.30	15.21	15.51	20.67	20.54
	40.10	39.42	18.45	18.96	5.10	5.02	15.71	15.04	20.64	21.56
	40.82	39.75	19.19	18.70	5.25	4.89	14.84	14.87	19.90	21.79
	40.48	40.06	18.71	18.90	5.21	4.86	14.65	15.20	20.95	20.98
	41.48	39.60	19.10	19.05	5.44	5.40	14.67	15.33	19.31	20.62
	41.29	39.95	18.80	19.32	5.28	5.33	14.72	14.90	19.91	20.50
Mean	40.74	39.97	18.84	19.19	5.21	5.09	14.85	14.98	20.36	20.79
Standard deviation	±0.56	±0.48	±0.25	±0.49	±0.13	±0.21	±0.37	±0.32	±0.60	±0.59

[a]Lead azide was obtained by difference from 100%.

[b]Barium nitrate was obtained by difference from 100%.

Table 4. NOL-130 Lone Star Batch 54

Table 5. NOL-130 Lone Star Batch 56.

	Lead styphnate		Lead azide		Tetracene		Antimony sulfide		Barium nitrate	
	Spec method	Polarograph	Spec method[a]	Polarograph	Spec method	Polarograph	Spec method	Polarograph	Spec method	Polarograph[b]
	41.05	40.92	19.22	19.19	5.02	4.96	15.05	14.87	19.66	20.06
	41.54	41.01	19.11	19.17	5.02	5.01	14.56	14.92	19.77	19.89
	41.26	41.30	19.16	19.32	5.13	4.89	14.80	14.79	19.65	19.70
	41.09	41.21	20.11	19.60	5.03	4.96	14.90	14.67	18.87	19.56
	41.37	40.80	19.56	19.21	4.96	5.02	14.90	14.99	19.21	19.98
	40.75	41.22	19.28	19.10	4.83	4.75	15.26	15.01	19.88	19.92
	41.41	41.04	19.30	18.98	4.95	4.96	14.82	14.88	19.52	20.14
	41.82	41.52	19.31	19.14	4.94	4.77	14.82	14.77	19.11	19.80
	41.35	41.05	18.90	19.33	5.01	5.10	15.20	15.10	19.54	19.42
	41.56	40.79	18.77	18.99	5.00	5.09	14.52	14.94	20.15	20.19
Mean	41.32	41.09	19.27	19.20	4.99	4.95	14.88	14.89	19.54	19.87
Standard deviation	±0.30	±0.23	±0.37	±0.18	±0.07	±0.12	±0.24	±0.13	±0.38	±0.25

[a]Lead azide was obtained by difference from 100%.

[b]Barium nitrate was obtained by difference from 100%.

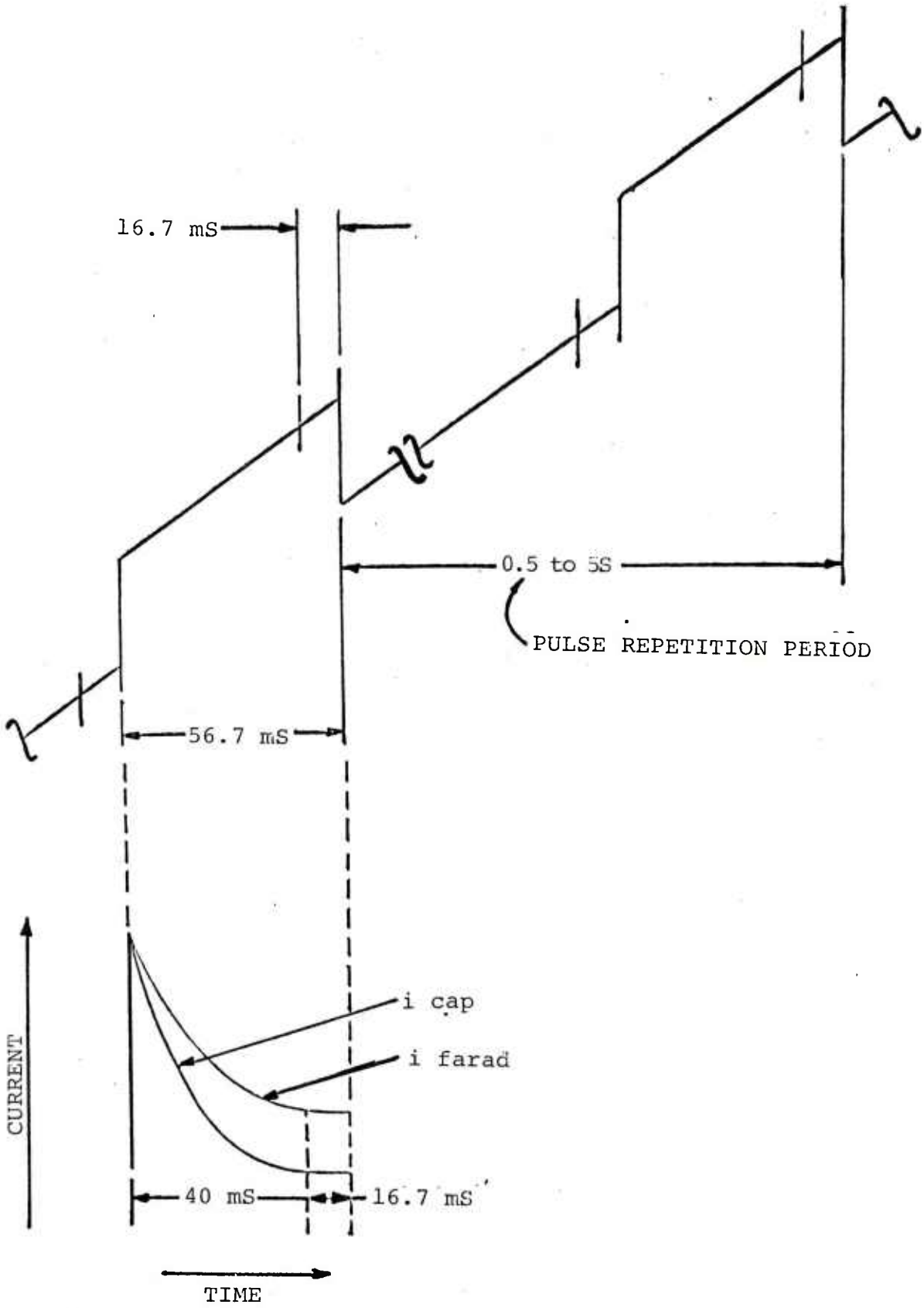

Figure 1. Differential pulse exitation wave form.

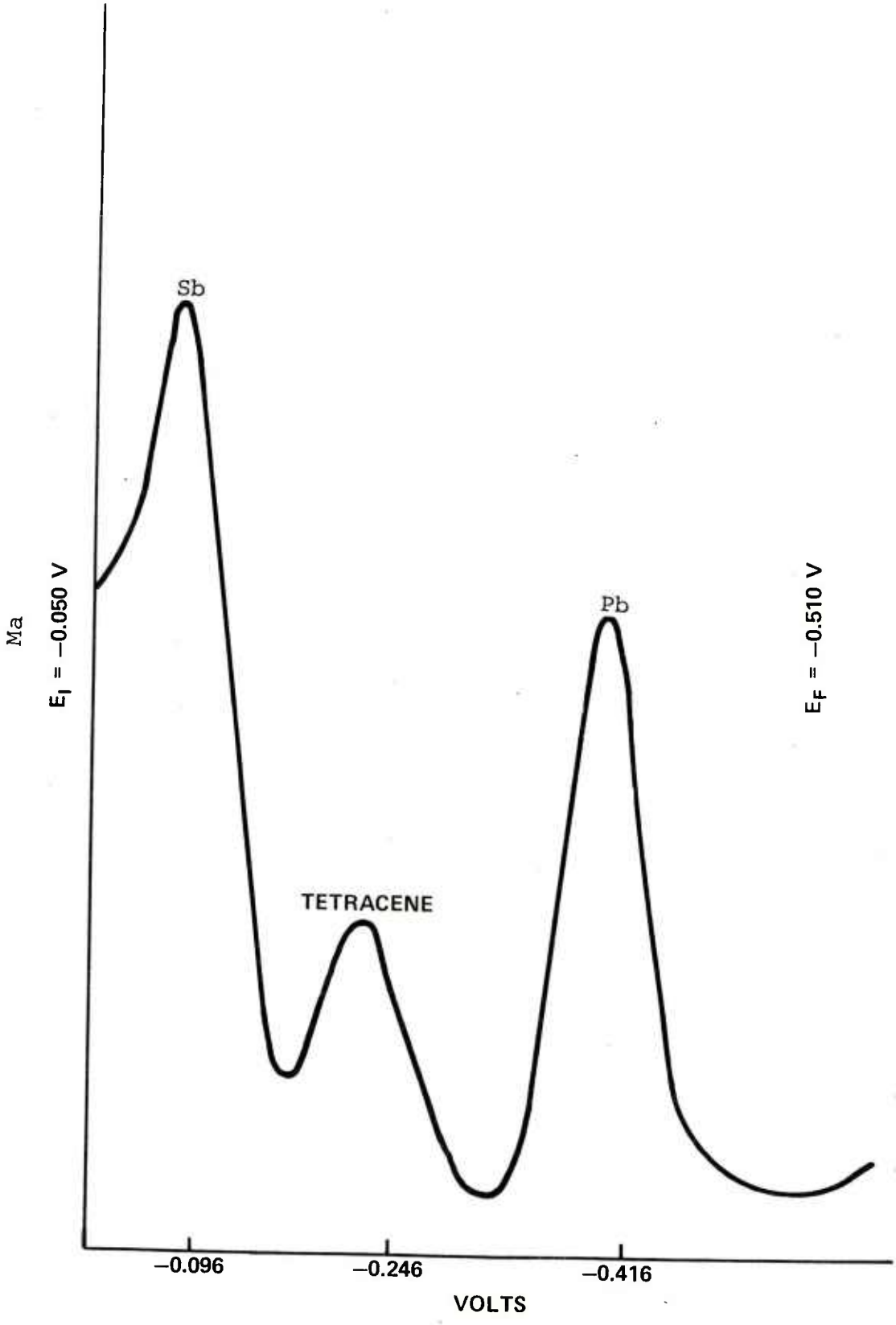

Figure 2. Polarogram of Sb, tetracene & Pb in 4M LiCl.

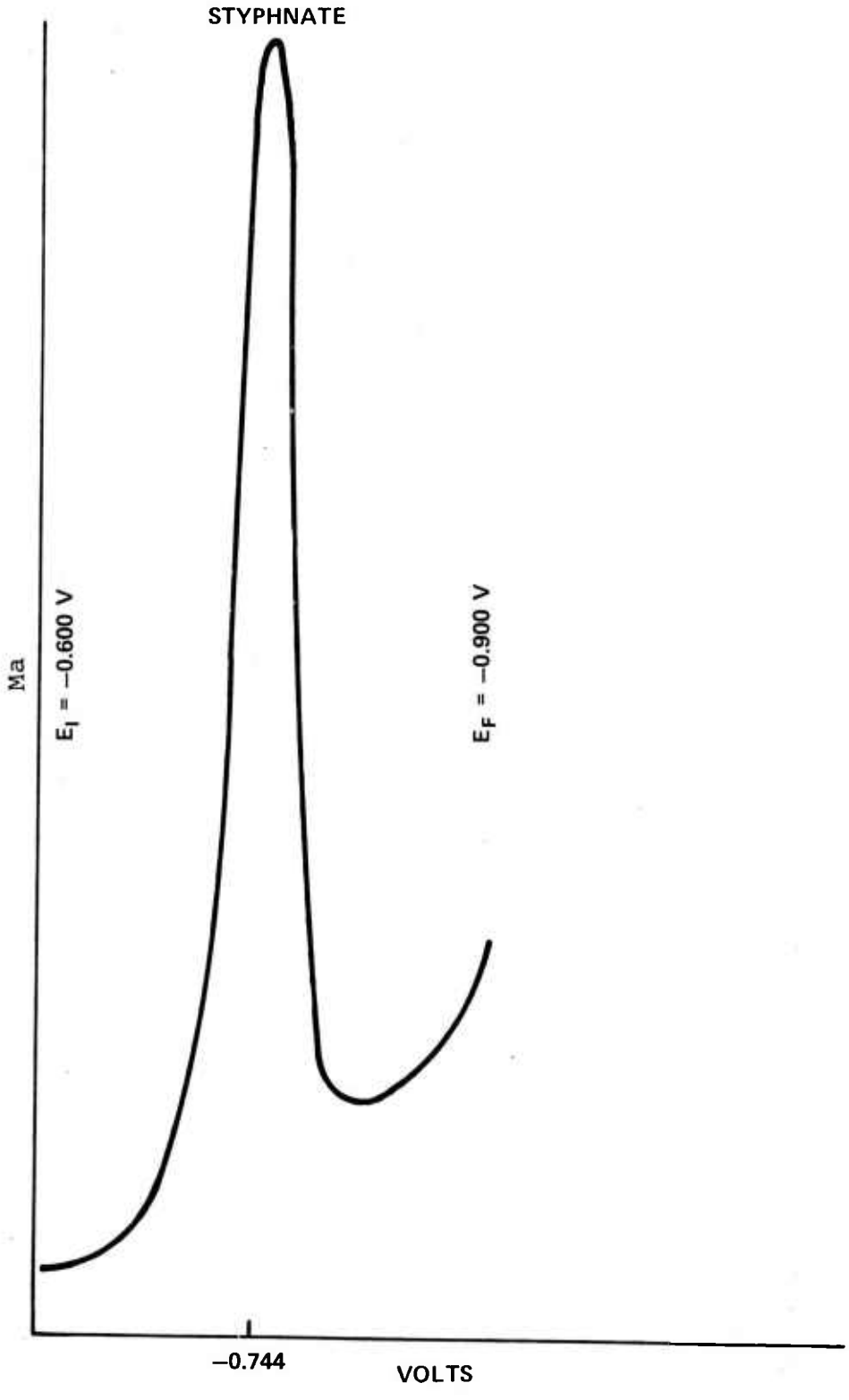

Figure 3. Polarogram of styphnate in 0.1M Tetra-n-butylammonium hydroxide.

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